



Department of Toxic Substances Control

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MEMORANDUM

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Data Review Of Perchlorate Test Results For Samples Collected At Bathtub SUBJECT:

Well #1 (OS-9), Brandeis Bardin, for Boeing Rocketdyne Project

(August 12, 2003)

A group of samples were collected or prepared at Bathtub Well #1 (OS-9), Brandeis Bardin, near the Boeing Rocketdyne Site on August 12, 2003. Where necessary, primary sample s were split to provide samples to more than one analyzing laboratory. The group of samples is described as follows:

- 1. Groundwater samples and groundwater sample duplicates
- 2. Groundwater samples spiked with perchlorate to levels of 5 ppb, 50 ppb and 150 ppb
- 3. Groundwater sample duplicates spiked with perchlorate to levels of 5 ppb, 50 ppb and 150 ppb
- 4. De-ionized water
- 5. De-ionized water samples spiked with perchlorate to levels of 5 ppb, 50 ppb and 150 ppb
- 6. Field blank
- 7. Groundwater samples spiked with perchlorate to levels of 5 ppb and 50 ppb at ambient temperature for stability study

The group of samples was analyzed for perchlorate by four different laboratories. They are: Del Mar Analytical (Del Mar), Ceimic Corporation (Ceimic), American Scientific Laboratories/Advanced Technology Laboratories (ASL/ATL), and the Hazardous Materials Laboratory-Southern California (HML-SC) of the Department of Toxic Substances Control (DTSC).

Samples analyzed by Del Mar and Ceimic were: groundwater sample, groundwater sample duplicate, groundwater sample spiked with perchlorate (5 ppb, 50 ppb, 150 ppb) groundwater sample duplicate spiked with perchlorate (5 ppb, 50 ppb, 150 ppb), de-ionized water, de-ionized water spiked with perchlorate (5 ppb, 50, 150 ppb) and field blank.

Samples analyzed by ASL/ATL were: groundwater sample, groundwater sample spiked with perchlorate (5 ppb, 50 ppb,150 ppb), de-ionized water, de-ionized water spiked with perchlorate (5 ppb, 50 ppb,150 ppb).

Samples analyzed by HML-SC were: groundwater sample, groundwater sample spiked with perchlorate (5 ppb, 50 ppb,150 ppb), de-ionized water, de-ionized water spiked with perchlorate (5 ppb, 50 ppb,150 ppb), and groundwater samples spiked with perchlorate to 5 ppb and 50 ppb at ambient temperature for stability study.

The samples analyzed by the four laboratories are summarized in Table 1. Table 1 shows the analyzing laboratory, sample identification number, perchlorate result and percent recovery for each sample spiked with known amount of perchlorate.

DATA EVALUATION

We have collected the data packages available from the four different laboratories. We evaluated the holding times, initial calibration (IC), instrument performance check (IPC), initial calibration check standard (ICCS), continuing calibration check standard(CCCS), method blank (MB), laboratory control sample/laboratory fortified blank (LCS/LFB), matrix spike/matrix spike duplicate (MS/MSD), perchlorate identification and quantitation according to the requirements of Method 314.0. Our review summary is given in Table 2.

Del Mar

Quality assurance/Quality Control (QA/QC) results were satisfactory and the reported sample results should be acceptable.

Ceimic

The data package has no IC raw data. The summary and calibration curve appeared to show acceptable initial calibration. Other QA/QC results were satisfactory. The reported sample results should be acceptable.

ASL/ATL

The sample run sequence for standards and samples is shown below. From the five point calibration standards, the average retention time (RT) for perchlorate is 9.70 min. The RT window would be 9.21 to 10.18 min. It appears that as the standard concentrations increase from 2 ppb to 100 ppb, the RT decreases from 9.97 min to 9.42 min. Also, the RT appears to decrease inversely to elapsed time. Thus, the 2 ppb standard had a RT of 9.97 min at the beginning of the sequence. However, the two 2 ppb standards indicate a RT of 9.58 min and 9.52 min after twelve sample analyses. Because of the RT shifting, the last six samples show RTs that are outside of the RT window. In some of the samples (ground water samples) there is a large peak eluted close to the perchlorate peak. This large unknown peak may contribute some interference to the accuracy of the perchlorate peak area counts. So, the analytical system used by ASL/ATL may not be stable and the perchlorate area counts may not be accurate. The reported results outside of the RT window should be qualified as estimates.

ASL/AT Samples	Retention Time	Perchlorate Concentration (ppb)
	(Minutes)	
Autocal1R		0
Autocal2R	9.97	2
Autocal3R	9.85	4
Autocal4R	9.78	10
Autocal5R	9.63	25
Autocal6R	9.52	50
Autocal7R	9.42	100
ICV 50	9.48	48.88
ICV25	9.55	26.57
ICB		0
IPC	9.43	25.7
MB		0
LCS	9.45	25.09
Std 2 ppb	9.58	2.15
Std 2 ppb	9.52	1.9
SSFLW236K	9.22	52.34 (Interference)
SSFLW238K	9.47	5.1
SSFLW239K	9.23	49.17
SSFLW241K		ND (Interference)
SSFLW245K		ND
SSFLW235K	9.30	5.4 (Interference)
SSFLW235KDUP	9.25	5.18 (Interference)
SSFLW237K(DF=2)	9.07 *	80.7 (Reported as 160) (Interference)
SSFLW240K (DF=2)	9.10 *	77.9 (Reported as 160) (Interference)
SSFLW235K-MS	9.17 *	13.95 (Interference)
SSFLW235K-MSD	9.17 *	14.58
CCV50	9.10 *	51.56
CCV25	9.20 *	25.61

Average RT for Autocal2R to 7R standards = 9. 7 min.

Interference = Unknown large peak close to the perchlorate peak

RT Window = 9.21 - 10.18 min.

^{*} RT outside RT window

HML-SC

Analyzed 8/13/03 (day 1): GW Sample, GW Samples Spike with Perchlorate to Levels of 5 ppb, 50 ppb, 150 ppb, deionized water (DI) Water and DI Water Spiked with Perchlorate to Levels of 5 ppb, 50 ppb, 150 ppb

EPA method 314.0 for the determination of perchlorate has many requirements. Section 9 and Table 6 discuss the quality control requirements, section 10 discusses the calibration and standardization requirements, section 11 discusses the procedure, and Table 7 provides an example sample analysis batch with quality control requirements.

The run sequence of standards and samples performed by HML-SC is shown below.

HML-SC Samples	Perchlorate Concentration (ppb)
Std 10 ppb	3.369 (34% Recovery)
SSFLW228K	0.447
SSFLW229K	51.83
SSFLW230K	177.181
SSFLW231K	6.388
SSFLW232K	59.477
Std 100 ppb	121.374 (121% Recovery)
SSFLW233K	180.636
SSFLW234K	0.194
SSFLW242K	0.315
SSFLW243K	55.257
SSFLW244K	0.294
2.5 ppb	0.626 (discarded)
5 ppb	1.657 (discarded)
10 ppb	5.633 (discarded)
25 ppb	23.297 *
35 ppb	31.847 (discarded)
50 ppb	47.535 *
70 ppb	71.258 *
100 ppb	85.949 (discarded)
150 ppb	147.16 *
200 ppb	203.065 *
Rinse	0.242
MB	0.345
MSR /LCS	53.456 (107% Recovery)
SSFLW231K-MS	56. 086 (100% Recovery)
SSFLW231K-MSD	56.027 (100% Recovery)
Std 5 ppb	6.167 * (123% Recovery)
Std 10 ppb	15.898 (159% Recovery)

^{*} Values of the standards used in initial calibration curve.

The run sequence, and calibration and standardization, do not conform with the method requirements. Standards should not be picked for initial calibration purposes.

Results of MSR or LCS, MB, MS/MSD were satisfactory. The average RT for perchlorate was 10.11 min. with a RT window of 9.6 – 10.62 min. The RTs of perchlorate detected in samples SSFLW229K, SSFLW230K, SSFLW231K, SSFLW32K,

SSFLW233K and SSFLW243K were within the RT window of 9.6 – 10.62 min. The QA/QC such as IPC, ICCS, and CCCS were not performed. As the data were generated without meeting the method requirements, all results should be qualified as estimates.

Analyzed 8/15/03 (Day 3): GW Samples Spiked with Perchlorate to Levels of 5 ppb and 50 ppb at Ambient Temperature for Stability Study

Standards 1 ppb, 2.5 ppb, 5 ppb, 10 ppb, 25 ppb, 50 ppb, 70 ppb and 100 ppb were analyzed. The laboratory discarded the lowest point (1 ppb) and middle point (10 ppb) and then constructed a linear calibration curve consisting of 2.5 ppb, 5 ppb, 25 ppb, 50 ppb, 70 ppb and 100 ppb. Generally, only the lowest or the highest standards can be dropped from a group of calibration standards. Thus, it is improper to discard the 10 ppb standard because it is not the lowest or the highest standard in a group of standards.

The QA/QC samples such as IPC, ICCS, CCCS, MB, MSR or LCS and MD/MSD were not performed. The average RT for perchlorate standards was 9.79 min. with a RT window of 9.3 -10.28 min. the RT of perchlorate reported in sample SSFLW243K was within the RT window. As the data were generated without meeting the method requirements, all results should be qualified as estimates.

Analyzed 8/26/03 (Day 14): GW Samples Spiked with Perchlorate to Levels of 5 ppb and 50 ppb at Ambient Temperature for Stability Study

Standards 2.5 ppb, 5 ppb, 10 ppb, 25 ppb, 50 ppb, 70 ppb and 100 ppb were analyzed. The laboratory discarded the lowest point (2.5 ppb) and middle point (10 ppb) and then constructed a linear calibration curve consisting of 5 ppb, 25 ppb, 50 ppb, 70 ppb and 100 ppb. As we discussed above, this is not an acceptable practice. No IPC, ICCS and CCCS were performed. Results of MB, MSR or LCS and MS/MSD were satisfactory. The average RT for perchlorate standards was 9.83 min with a RT window of 9.34 – 10.32 min. The RT of perchlorate reported for sample SSFLW243 was within the RT window of 9.34 -10.32 min.

Sample SSFLW242 K (8/26/03) was reported as ND with a reporting limit/quantitation limit (RL/QL) of 2.5 ppb. Since 2.5 ppb was discarded from the linear calibration curve, 5 ppb become the lowest standard. The RL/QL should be 5 ppb and not 2.5 ppb as reported for sample SFLW242K.

As the data were generated without meeting the method requirements, all results should be qualified as estimates.

Thus, all HML-SC reported perchlorate results should be qualified as estimates.

GW, Spiked GW, DI-Water and Spiked DI-Water Samples Results

All reported results by the four laboratories are given in Table 1. The results reported by Del Mar and Ceimic agree very well. Some of the results reported by ASL/ATL were outside of the established retention time window and should be qualified as estimates. The results reported by HML-SC should all be qualified as estimates and have mostly a high detection limit (5 ppb)

compared to the detection limits reported by other laboratories (less than 4 ppb). However, the overall reported results by the four laboratories are consistent and within the same order of magnitude.

HML-SC Ambient Temperature Stability Study

For GW spiked with 5 ppb perchlorate, reported results of ND (5) analyzed on 8/13/03 (day 1), ND (2.5) analyzed on 8/15/03 (day 3) and ND (2.5) analyzed on 8/26/03 (day 14) should be only estimates. Therefore, stability of perchlorate at 5 ppb spike at ambient temperature can not be determined.

For GW spiked with 50 ppb perchlorate, reported results of 55 ppb analyzed on 8/13/03(day1), 54 ppb analyzed on 8/15/03 (day 3) and 53 ppb analyzed on 8/26/03 (day 14) appear to be consistent. Recoveries of 106% - 110% are reasonable. Even though the reported results are qualified as estimates, it appears that a concentration of 50 ppb perchlorate in GW is stable up to 14 days at ambient temperature.

If you have any questions, please contact me or Lorna Garcia at (510)540-3003.

Cc: Bart Simmons, Ph.D.

Cindy Dingman Lorna Garcia James Cheng Rustum Chin Hamik Babian

Table 1: Summary of Perchlorate Test Results for Samples Collected at Bathtub Well #1 (OS-9)
August 12, 2003

		PERCHLORATE RESULTS and % RECOVERY										
Type of Sample	Del Mar Sample ID	Ug/L	Spike % R	Ceimic Sample ID	Ug/L	Spike % R	ASL/ATL Sample ID	Ug/L	Spik e % R	HML-SC Sample ID	Ug/L	Spike % R
Groundwater (GW) Sample	OS09081203-01	ND (4)		OS09081203-03	ND (1)		SSFLW241K	ND (1)		SSFLW234K	ND (5)*	
GW 5 ppb Spike	OS09081203-6M1	4.2 (4)	84	OS09081203-6M3	4.51 (1)	90.20	SSFLW235K	5.4(2)	108	SSFLW 228K	ND (5)*	0
GW 50 ppb Spike	OS09081203-6MB1	49	98	OS09081203-6MB3	49.80	99.60	SSFLW236K	52	104	SSFLW229K	52*	104
GW 150 ppb Spike	OS09081203-06MC1	150	100	OS09081203-06MC3	141.43	94.29	SSFLW237K	160	107	SSFLW230K	180*	120
GW Sample Duplicate	OS09081203-02	ND (4)		OS09081203-03D	ND (1)		NA	NA		NA	NA	NA
GW Duplicate 5 ppb Spike	OS09081203-06MD1	4.4 (4)	88	OS09081203-06MD3	4.48 (1)	89.60	NA			NA	NA	NA
GW Duplicate 50 ppb Spike	OS09081203-06MBD1	49	98	OS09081203-06MBD3	49.91	99.82	NA			NA	NA	NA
GW Duplicate 150 ppb Spike	OS09081203-06MCD1	150	100	OS09081203-06MCD3	140.07	99.38	NA	NA	NA	NA	NA	NA
DI Water	OS09081203-06N1	ND (4)		OS09081203-06N3	ND (1)		SSFLW245K	ND (2)		SSFLW244K	ND (5)*	
DI Water 5 ppb Spike	OS09081203-06R1	4.6 (4)	92	OS09081203-06R3	4.62(1)	92.40	SSFLW238K	5.1(2)	102	SSFLW231K	6 (5)*	120
DI Water 50 ppb Spike	OS09081203-06RB1	49	98	OS09081203-06RB3	49.53	99.06	SSFLW239K	49	98	SSFLW232K	59*	118
DI Water 150 ppb Spike	OS09081203-06RC1	150	100	OS09081203-06RC3	141.71	94.70	SSFLW240K	160	107	SSFLW233K	180*	120
Field Blank	OS09081203-004	ND (4)		OS09081203-03F	ND (1)		NA	NA	NA	NA	NA	NA
GW 5 ppb Spike Ambient Temperature	NA	NA	NA	NA	NA	NA	NA	NA	NA	SSFLW242K	ND (5) * ND (2.5)**	0
Stability Study											ND (2.5)***	0
GW 50 ppb Spike Ambient Temperature	NA	NA	NA	NA	NA	NA	NA	NA	NA	SSFLW243K	55* 54**	110 108
Stability Study											53***	106

 $HML\text{-}SC = Hazardous \ Materials \ Laboratory\text{-}Southern \ California$

ND = Non-Detect

% R = Percent Recovery of spike sample

Ambient Temperature Stability Study:

* = analyzed 8/13/03 (Day 1)

** = analyzed 8/15/03 (Day 3)

*** = analyzed 8/26/03 (Day14)

ASL/ATL = American Scientific Laboratory/Advanced Technology Laboratories

NA = Not Analyzed

() = Reporting Limit/Quantitation Limit

DI Water = De-ionized Water

Table 2: Perchlorate Data Review QA/QC Summary

QA/QC and Acceptance Criteria	ACCEPTABILITY					
	Del MAR	Ceimic	ASL/ATL	HML-SC		
	(QC/QC Result)	(QC/QC Result)	(QC/QC Result)	(QC/QC Result)		
Holding Times 28 days	YES	YES	YES	YES		
Instrument Performance Check (IPC) Recovery = 80% - 120%	YES	YES	YES	Not Performed		
	(101%)	(93%, 90%)	(103%)			
Initial Calibration (IC) Correlation Coefficient = 0.99	YES	YES	YES	NO ^a		
	(0.9997)	(0.9999)	(0.9999)			
Initial Calibration Check Standard (ICCS)	YES	YES	YES	Not Performed		
Recovery = 75% - 125%	(105%)	(92%, 95%)	(97.8%)			
Continuing Calibration Check Standard (CCCS)	YES	YES	YES	Not Performed		
Recovery = 85% - 115%	(99% - 104%)	(94% - 98%)	(102% - 106%)			
Method Blank = 1/2 MRL</td <td>YES</td> <td>YES</td> <td>YES</td> <td>YES*,*** Not performed**</td>	YES	YES	YES	YES*,*** Not performed**		
Laboratory Control Sample/ Laboratory Fortified Blank	YES	YES	YES	Not Performed ** YES *, ***		
Recovery = 85% - 115%	(100%)	(94%)	(100%)	(MSR or LCS=107%, 86%)		
Matrix Spike/ Matrix Spike Duplicate Recovery = 80% - 120% RPD = +/- 15%	YES	Not Performed	YES	YES		
	(96%, 97% R) (1% RPD)		(92%, 86%) (7% RPD)	(102%,102%R, 0%RPD)* (Not performed)** (117%,101% R,15%RPD)***		
Identification	YES	YES	YES	YES		
Quantitation	YES	YES	YES	Results qualified as estimates		

ASL/STL = American Scientific Laboratory/Advanced Technology Laboratories

HML-SC = Hazardous Material Laboratory-Southern California

Ambient Temperature Stability Study

* Analyzed 8/13/03 (day 1)

** Analyzed 8/15/03 (day 3)

*** Analyzed 8/26/03 (day 14)

a Not performed according to method requirements.